Remarks

The Office Action mailed 15 July 2003 has been received and reviewed. Claim 20 having been amended, the pending claims are claims 1-11, 13-15, and 17-32. Claims 16, 31, and 32 have been withdrawn from consideration by the Examiner.

Claim 20 has been amended to recite "less than or equal to about 45 atomic percent oxygen," which is supported, for example, by claim 1.

Reconsideration and withdrawal of the rejections are respectfully requested.

Examiner's Interview

Applicants thank the Examiner for granting a telephonic interview to Applicants' Representative, Loren D. Albin, on 17 September 2003.

The status of the claims was discussed, and Applicants' Representative pointed out that the Office Action mailed 15 July 2003 does not list claims 16, 31, and 32 as pending. Although claims 16, 31, and 32 were withdrawn from consideration by the Examiner in prior actions, Applicants have not canceled claims 16, 31, and 32. Agreement was reached that claims 16, 31, and 32 are pending, as reflected in the status of the claims herein above.

Applicants' representative reiterated arguments already of record for the patentability of the presently pending claims over the cited art. No agreement was reached, but the Examiner agreed to reconsider the rejections upon submission of a Declaration under 37 C.F.R. §1.132.

Rejection under 35 U.S.C. §102

The Examiner rejected claims 1-11, 13-15, 17-25, and 27-30 under 35 U.S.C. §102 (b, b, e, e,) as being anticipated by WO 98/33948, WO 98/59089, Dorfman et al. (U.S. Patent No. 5,352,493), or Neerinck et al. (U.S. Patent No. 6,228,471 B1). The Examiner alleged that the documents all teach the claimed diamond film coated on an object. Applicants respectfully traverse the rejection.

Applicants respectfully assert that each of the cited documents fails to teach the presently claimed fluid handling devices that include a dense random covalent diamond-like glass (DLG). Support for the assertion is provided by the Declaration under 37 C.F.R. §1.132 by Moses M. David, Ph.D., an inventor of the present application.

Specifically, the Declaration states that the cited documents each

> describe their materials as two interpenetrating networks, rather than the claimed random covalent system. For example, Figures 1A, 1B, and 1C in Dorfman clearly show that the carbon in the a-C:H network is only bonded to either carbon or hydrogen, and the silicon in the a-Si:O network is only bonded to either silicon or oxygen. This precludes the covalent bonding configurations such as Si-C, Si-H, O-H and C-O present in a random covalent network. Such interpenetrating networks would not be random covalent network as recited in the claims in the present 09/519,448 application. (Paragraph 15 of the Declaration under 37 C.F.R. §1.132 submitted herewith).

Further, Applicants respectfully assert that the materials disclosed in the cited documents have different physical properties than the DLG disclosed in the present application. Support for the assertion is provided by the Declaration under 37 C.F.R. §1.132 by Moses M. David, Ph.D., an inventor of the present application. Specifically, the Declaration states that the cited documents each fail to teach a film that necessarily imparts low fluorescence (e.g., claims 27 and 28) or necessarily imparts a hydrophilic surface (e.g., independent claims 18, 19, 25, 28, and 30).

In view of the remarks presented herein above, Applicants respectfully request that the Examiner reconsider and withdraw the rejection under 35 U.S.C. §102.

Restriction Requirement and Request for Rejoinder

The claims that have been withdrawn from consideration (e.g., claims 26, 31, and 32) include all the claim language of claims 18, 28, and 30, respectively. Applicants respectfully request that the Restriction Requirement be reconsidered and withdrawn.

In the event that the Examiner maintains the Restriction Requirement and pursuant to M.P.E.P. §821.04, rejoinder of the non-elected method claims is respectfully requested upon notice of allowance of any of the elected product claims. *See, also, In re Ochiai*, 37 USPQ2d 1127 (Fed. Cir. 1995) and *In re Brouwer*, 37 USPQ2d 1663 (Fed. Cir. 1996).

Summary

It is respectfully submitted that all the pending claims are in condition for allowance and notification to that effect is respectfully requested. The Examiner is invited to contact Applicants' Representatives, at the below-listed telephone number, if it is believed that prosecution of this application may be assisted thereby.

Respectfully submitted for
Moses M. DAVID et al.
By
Mueting, Raasch & Gebhardt, P.A.
P.O. Box 581415
Minneapolis, MN 55458-1415
Phone: (612) 305-1220
Facsimile: (612) 305-1228

October 15, 2003
Date

By: _____
Loren D. Albin
Reg. No. 37,763
Direct Dial (612)305-1225

CERTIFICATE UNDER 37 CFR §1.8:
The undersigned hereby certifies that the Transmittal Letter and the paper(s), as described hereinabove, are being transmitted by facsimile in accordance with 37 CFR §1.6(d) to the Patent and Trademark Office, addressed to Assistant Commissioner for Patents, Mail Stop AF, P.O. Box 1450, Alexandria, VA 22313-1450, on this 15th day of October, 2003, at 1:17 p.m. (Central Time).

By: _____
Name: Rachel L. Eaglesdi-Graham

PATENT
Docket No. 55436US002

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant(s):	Moses M. DAVID et al.	Group Art Unit:	1743
Serial No.:	09/519,448	Examiner:	Lyle Alexander
Confirmation No.:	6966		
Filed:	5 March 2000		
For:	FLUID HANDLING DEVICES WITH DIAMOND-LIKE FILMS		

Declaration under 37 C.F.R. §1.132

Assistant Commissioner for Patents
Mail Stop AF
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

I, Moses M. David, Ph.D., declare as follows:

1. I am an inventor of the above-identified patent application Serial No. 09/519,448. I received a Bachelor's degree in Chemical Engineering from Indian Institute of Technology, Madras, India, in 1985; a Master's degree in Chemical Engineering from McGill University in Montreal, Canada, in 1987; and a Doctorate in Chemical Engineering from Clarkson University in Potsdam, New York, in 1990. I am an employee of 3M Company, and have been employed by 3M Company for more than 12 years. My current position is that of Senior Research Specialist.

2. I have authored 12 scientific publications, at least 8 of which are in the field of diamond-like films. One of my publications is attached as Exhibit 1, (i.e., David et al., "Diamond-Like Film-Encapsulated Fibers Enable Long-Length Grating Production," Lightwave, July, 2000). Diamond-like glass (DLG) materials have also been recognized as important

developments in winning a 2002 Photonics Circle of Excellence Award (see Exhibit 2). I have 15 years of experience in the field of diamond-like films deposited by vacuum processes.

3. I have read and understood the Office Action mailed January 31, 2003 in regard to patent application Serial No. 09/519,448.

4. The coinventors and I have invented a fluid handling device that includes DLG. The DLG includes a dense random covalent system including on a hydrogen-free basis at least about 30 atomic percent carbon, at least about 25 atomic percent silicon, and less than or equal to about 45 atomic percent oxygen. Certain preferred embodiments of the invention may include optically transmissive films including DLG, which more preferably are at least 50 percent transmissive to radiation at one or more wavelengths from about 180 nm to about 800 nm. Certain preferred embodiments of the invention may include hydrophilic films including DLG. Additionally, in certain preferred embodiments, the DLG films of the present invention exhibit substantially no fluorescence.

5. On information and belief, the crystallinity and the nature of the bonding of the carbon network determine the physical and chemical properties of the deposited DLG made in accordance with the invention claimed in patent application Serial No. 09/519,448. Even if two materials have similar ingredients, they can have significantly different properties if the ingredients are combined under differing conditions. For example, if two materials have differing networks or differing chemical bonds, then the physical properties of the two materials can be dramatically different. As acknowledged in the specification of patent application Serial No. 09/519,448, materials have previously been modified with diamond-like thin films having significantly different properties from the DLG of the present invention. The differences in properties are believed to be due, at least in part, to the arrangement and bonding f atoms in the

thin films. The unique characteristics of the DLG disclosed in patent application Serial No. 09/519,448 include the presence of at least about 30 atomic percent carbon, at least about 25 atomic percent silicon, and less than or equal to about 45 atomic percent oxygen or a hydrogen free basis formed into a dense random covalent system.

6. The above-referenced patent application Serial No. 09/519,448 includes an example at pages 24-25 that establishes that the presently disclosed DLG imparts low fluorescence on glass capillaries. The low fluorescence of capillaries having DLG made in accordance with the teachings of patent application Serial No. 09/519,448 disposed thereon, provides for capillaries that exhibit substantially no fluorescence when exposed to the light used to irradiate a target species in the instance where excitation is effected through the capillary wall. Such a capillary, for instance, does not require the film to be removed for optical detection of the samples.

7. The above-referenced patent application Serial No. 09/519,448 includes an example at pages 25-26 that establishes that the presently disclosed DLG can impart a hydrophilic surface on glass capillaries. The above-referenced patent application Serial No. 09/519,448 defines "hydrophilic" to mean a film having a water contact angle of about 50 degrees or less (page 5, lines 6-8). The hydrophilic surface of glass capillaries having DLG made in accordance with the teachings of patent application Serial No. 09/519,448 disposed thereon, provides for capillaries that have good wetting and flow characteristics.

8. I have read and am familiar with PCT International Publication Number WO 98/59089 to N.V. Bekaert S.A., entitled "Method for making a non-sticking diamond-like nanocomposite." This patent is hereafter referred to as "Bekaert."

9. I have read and am familiar with PCT International Publication Number WO 98/33948 and U.S. Pat. No. 6,228,471 to N.V. Bekaert S,A,, both titles reciting a "coating comprising layers of diamond like carbon and diamond like nanocomposite compositions." These documents are hereinafter collectively referred to as "Bekaert 2."

10. I have read and am familiar with U.S. Pat. No. 5,352,493 to Dorfman et al., entitled "Method for forming diamond-like nanocomposite or doped-diamond-like nanocomposite films". This patent is hereafter referred to as "Dorfman".

11. In my opinion, Bekaert, Bekaert 2, and Dorfman each fail to teach the claimed fluid handling devices that include a dense random covalent diamond-like glass (DLG).

12. Bekaert discloses a "non-sticking diamond-like nanocomposite composition comprising networks of a-C:H and a-Si:O" (Abstract). "Diamond Like Nanocomposite (DLN) compositions consist of an amorphous random carbon network which is chemically stabilized by hydrogen atoms. The carbon *network is interpenetrated* with an amorphous glass-like *silicon network* which is chemically stabilized by oxygen atoms (a-C:H/a-Si:O)" (page 1, lines 14-18; emphasis added). The "Diamond Like Nanocomposite (DLN) compositions" disclosed by Dekaert are not the *diamond-like glass* including a dense random covalent system as recited in the present claims.

13. Bekaert 2 discloses "layered structures . . . comprising a first diamond like nanocomposite composition layer . . . which . . . comprises *interpenetrating networks* of a.C:H and a.Si:O, a second diamond like carbon composition layer . . . , a transition layer . . . comprising a mixture of said diamond like nanocomposite and said diamond like carbon composition." (Abstract; emphasis added). The "diamond like nanocomposite composition"

disclosed by Bekaert 2 is not the *diamond-like glass* including a dense random covalent system as recited in the present claims.

14. Dorfman discloses "the formation of a class of nanocomposite amorphous materials consisting of *interpenetrating random networks* of predominantly sp3 bonded carbon stabilized by hydrogen, glass-like silicon stabilized by oxygen and random networks of elements from the 1-7*b* and 8 groups of the periodic table." (Abstract, emphasis added). The "interpenetrating random networks" disclosed by Dorfman is not the *diamond-like glass* including a dense random covalent system as recited in the present claims.

15. Bekaert, Bekaert 2, and Dorfman each fail to teach a dense random covalent DLG material as disclosed in the above-referenced patent application Serial No. 09/519,448. In particular, Bekaert, Bekaert 2, and Dorfman each describe their materials as two interpenetrating networks, rather than the claimed random covalent system. For example, Figures 1A, 1B, and 1C in Dorfman clearly show that the carbon in the a-C:H network is only bonded to either carbon or hydrogen, and the silicon in the a-Si:O network is only bonded to either silicon or oxygen. This precludes the covalent bonding configurations such as Si-C, Si-H, O-H and C-O present in a random covalent network. Such interpenetrating networks would not be random covalent network as recited in the claims in the present 09/519,448 application.

16. On information and belief, the differences in the materials used in Bekaert, Bekaert 2, and Dorfman, along with the way in which they are combined, and the physical properties of the resulting material, make these materials quite different from the DLG that has been disclosed in patent application Serial No. 09/519,448.

17. In my opinion, Bekaert, Bekaert 2, and Dorfman each fail to teach a film that necessarily imparts low fluorescence on glass capillaries. Notably, Bekaert, Bekaert 2, and Dorfman are silent regarding low fluorescence.

18. In my opinion, Bekaert, Bekaert 2, and Dorfman each fail to teach a film that necessarily imparts a hydrophilic surface on glass capillaries. In fact, Bakaert states that "[t]he contact angle of a water droplet on a surface coated with the DLN composition according to the proposed method, has been measured to be 90 to 95°." (Page 5, lines 7-9). Similarly, Bakaert 2 states that "[a]n increase in Si and O will thereby increase the contact angle and thereby generally also improve the coating's aptitude as a release or non-sticking coating or as a barrier layer against humidity." (Column 6, lines 60-63 of U.S. Pat. No. 6,228,471).

19. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the likes so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

October 14, 2003
Date

Moses M. David
Moses M. David, Ph.D.

EXHIBIT 1

LIGHTWAVE special reports

Diamond-like film-encapsulated fibers enable long-length grating production

A new method of fiber Bragg grating production preserves the mechanical integrity of the fiber and produces higher yields.

Moses M. David and James F. Brennan, III
3M

Several researchers have been perfecting the fabrication of fiber Bragg gratings that are several meters in length.[1-3] The application of these devices in optical telecommunications systems is imminent. A great deal of theoretical and experimental work has been done to understand the optical performance of these devices, but until recently, little attention has been given to maintaining the mechanical integrity of the gratings. Before they are seriously considered for use in communications systems, long fiber gratings must pass rigorous mechanical reliability tests and be easily manufactured.

In standard fiber Bragg grating production, the protective coating on a fiber must be removed before a grating can be written. Afterward, the fiber is immediately recoated to preserve its mechanical strength. The process of stripping and recoating a fiber is time-consuming and tedious, requiring skilled technicians. Additionally, the bare fiber is prone to damage during grating inscription, which reduces production yields. These problems are exacerbated when long lengths of fiber are stripped, handled, and recoated.

Several solutions have been proposed to eliminate these process steps. Most of the solutions involve writing gratings through specialized, UV-transparent polymeric fiber coatings.[4-6] Writing gratings through a fiber coating may be a solution for short-length (less than a few centimeters) fiber Bragg grating production, but t be used in long-length grating production, a write-through coating must meet demanding physical and optical requirements.[7]

A process has recently been developed to deposit a UV-transparent, diamond-like film onto fiber to maintain the mechanical integrity of the fiber close to that of pristine fiber. By depositing a thin film on the fiber (~1-5 microns thick), fiber geometry such as concentricity and diameter is preserved to the degree necessary to produce long fiber Bragg gratings. This diamond-like film can also withstand the temperatures used to anneal fiber gratings.

Carbon films contain substantially two types of carbon-carbon bonds: trigonal graphite bonds (sp2) and tetrahedral diamond bonds (sp3). Diamond is composed almost entirely of tetrahedral bonds; diamond-like film, approximately 50% to 90% tetrahedral bonds; and graphite, nearly all trigonal bonds. The crystallinity and nature of the bonding of the carbon network can be manipulated to affect the physical and chemical properties of a diamond-like film. Diamond is crystalline and essentially pure carbon, whereas diamond-like film is amorphous and can contain a substantial amount of additional components.

The composition of diamond-like films can be tailored so they have desirable diamond-like optical and mechanical properties such as extreme hardness (1,000 to 2,000 kg/mm^2 Vickers). Diamond's optical bandgap is 5.56 eV (~223 nm) and is therefore transmissive in the ultraviolet, but the polycrystalline structure of diamond films causes light scattering from grain boundaries. Diamond-like films are amorphous and don't suffer from light scattering at grain boundaries. The carbon-carbon d uble-bonding prevalent in diamond-like film causes undesirable optical absorption below ~500 nm.

By incorporating silicon and oxygen atoms into the amorphous diamond-like carbon film during the deposition process, absorption is altered. For example, diamond-like film of 1 micron thick was deposited on quartz slides as the oxygen flow rate during deposition was varied, and the ptical transmissions through these slides were measured (see Figure 1).

In the visible wavelength region, the optical transmission is close to 100% for all diam nd-like films, irrespective of the chemistry. In the UV region, dia-

LIGHTWAVE special reports

mond-like films' transmission drops dramatically when the oxygen flow falls below 10 sccm (standard cubic centimeters per minute). The optical transmission of the 10-sccm film drops below 30% at 244 nm, a commonly used wavelength for writing Bragg gratings. Under certain deposition conditions, a 1-micron-thick layer of diamond-like film is ~98% transmissive at 244 nm. The oscillations in the transmission spectra are due to thin-film interference.

The electronic-absorption edges in diamond-like films follow the Tauc relation:

$$\sqrt{\alpha E} = G(E - E_0),$$

where G is a constant, E_0 the optical gap, E the photon energy, and α the absorption coefficient.[8] The optical gap is taken from the extrapolation of the linear portion of the curves toward the abscissa. Tauc plots were calculated from data like that shown in Figure 1, and the optical gap was seen to change with oxygen content from −4.1 to −4.8 eV (see Figure 2). The chemical composition of the diamond-like films changed monotonically, even though abrupt changes occurred in their optical properties.

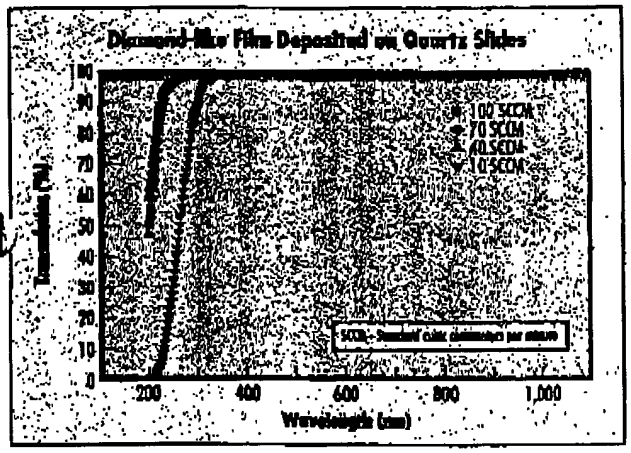

Figure 1. Optical transmission through 1-micron-thick layers of diamond-like films as a function of oxygen flow rate during deposition.

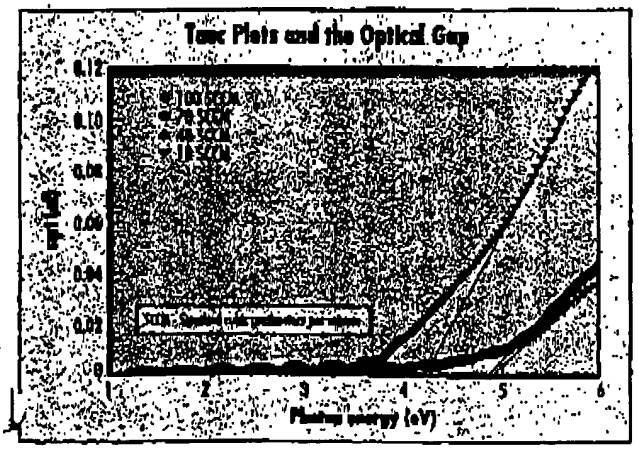

Figure 2. Tauc plots of diamond-like films made with varying oxygen flow rates.

Diamond-like film-encapsulated fibers

To deposit diamond-like film onto fibers, a capacitively coupled reactor system is used with two electrodes in a reaction chamber (see Figure 3). During deposition, the chamber is partially evacuated, and power at radio frequencies is applied to the electrodes. A carbon-containing source is introduced between the electrodes to form plasma comprising reactive species in proximity to the electrodes as well as form an ion sheath proximate to the electrodes. A fiber-handling apparatus is also used, where a fiber is stripped of its acrylate coating with a continuous-flow acid bath before it enters the reaction chamber and is drawn through the reactive ion sheath at a controlled speed. A uniform thickness film of ~5 microns was deposited onto the fiber, as shown in the micrograph in Figure 4. The variations in film thickness along the fiber were immeasurable but are estimated to be <10 nm.

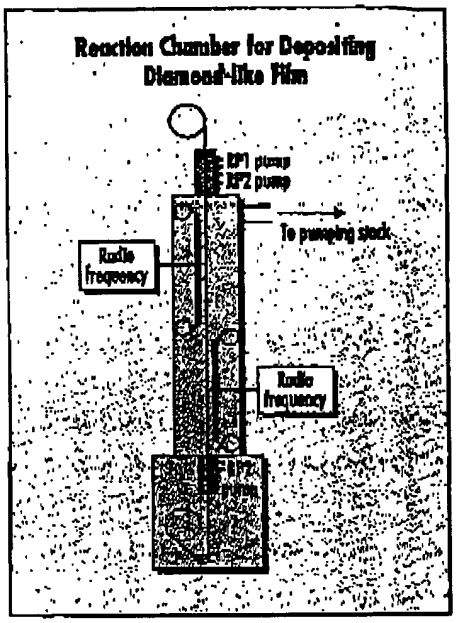

Figure 3. Schematic of chamber for depositing diamond-like film onto optical fiber. After it is evacuated with the pumps (RP1, RP2), power is applied at radio frequencies to the electrodes.

The mechanical properties of diamond-like film-encapsulated fibers were compared with those of acrylate-coated fibers and uncoated fibers. The diamond-like film-encapsulated fibers were insensitive to handling, such as by wiping the fibers between fingers or wrapping them on mandrels. In contrast, stripped fibers easily broke under such handling. As shown in Figure 5, the probability of fracture for both the acrylate-coated and diamond-like-film fibers were similar and occurred at a much higher tensile force than that for uncoated fibers.

Gratings were fabricated in the diamond-like film-encapsulated fiber with a frequency-doubled Ar^+ laser (244 nm) and compared to gratings written in standard fibers under identical conditions. As shown in Figure 6, there was no significant difference in the quality or reflectivity between the gratings. Visual inspection under a microscope discerned no signs of physical damage to the fiber surface. Long-length gratings (>10 cm) were fabricated in these fibers with a previously detailed method[2], and there was

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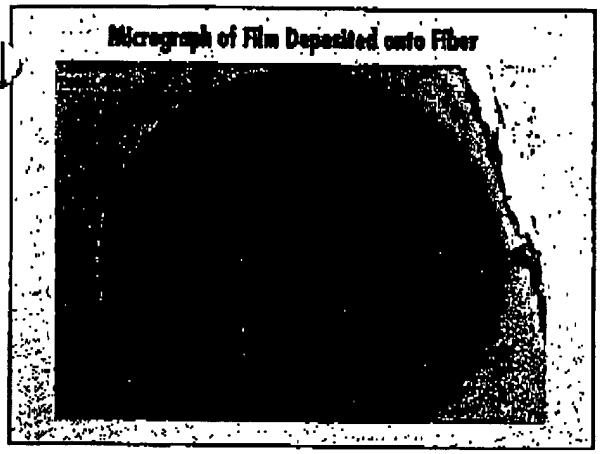


Figure 4. Micrograph of optical fiber protected with ~5-micron-thick diamond-like film.

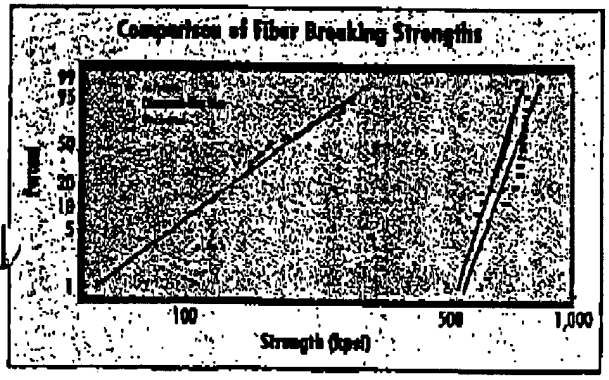


Figure 5. Breaking strength after handling optical fibers with diamond-like-film protection, acrylate coatings, and no coating.

no noticeable degradation in grating quality compared to gratings written in bare fiber. Any deviations that may have been caused by the diamond-like film are within the current limitations of the long-length fiber Bragg grating fabrication system. The mechanical strengths of these long-length gratings were then tested. Results similar to those in Figure 4 were obtained. These fibers were annealed at 100°C for 18 hours, and no mechanical strength degradation was observed.

Diamonds in the rough

These findings present significant opportunity for the fabrication of long-length fiber Bragg gratings that lend themselves to high-yield manufacturing processes. Critical to the process is this UV-transparent film, which protects the fiber during fiber grating inscription and maintains the geometry of the fiber during fabrication.

By developing a process to deposit a uniform layer of diamond-like film onto optical fibers, many of the major process and handling obstacles have been overcome. This significant new process opens up opportunity for further applications of longer-length fiber Bragg gratings throughout the optical network. ◆

References

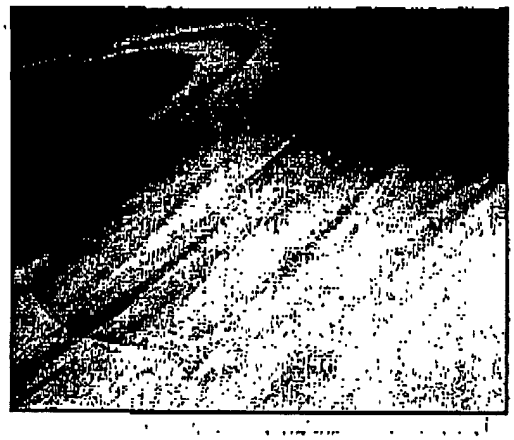
1. H. Rourke, et al., BGPP '99, ThD1.
2. J. Brennan and D. LaBrake, BGPP '99, ThD2.
3. L. Garrett, et al., OFC '99, PD15.
4. R. Espindola, et al., OFC '97, PD4.
5. K. Imamura, et al., Electron. Lett., 1998, 34 (10), 1016-7.
6. L. Chao and L. Reekie, OFC '99, ThD5.
7. M. Ibsen and R Laming, OFC '99, FA1.
8. J. Mort and F. Jansen, "Plasma Deposited Thin Films" (CRC Press, Boca Raton, 1986), 111-4.

Moses David, Ph.D., is an advanced research specialist at 3M (St. Paul, MN), where he specializes in plasma processing, diamond-like films, and thin-film optics. James F. Brennan, III, Ph.D., is a research specialist for 3M Telecom Systems Division (Austin, TX), where he works on the fabrication of long-length gratings for dispersion compensation.

EXHIBIT 2

The 2002 Photonics Circle of Excellence Award Winners

3M Co.
Film and Light Management
• UV Reflection Filters •



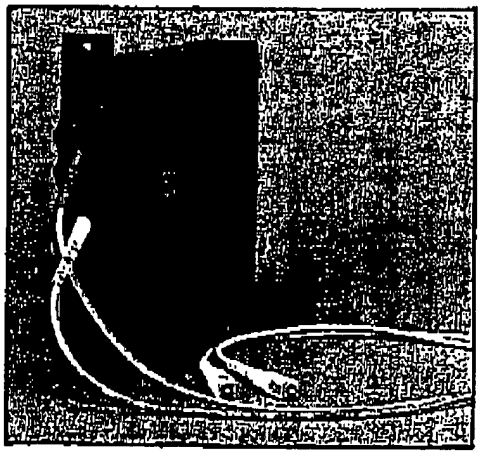
Thin, high-performance UV-reflective filters from 3M are constructed from engineering-grade polymers and do not require a substrate. Based on the St. Paul, Minn., company's Multilayer Optical Film technology, the flexible filters are 20 to 50 μm thick and eliminate the need to bond components individually. The films typically comprise several hundred layers of two polymers, each with its own light-refractive properties.

Suitable for high-end applications, the multilayer polymer filters can be structured to provide a sharp band edge transition between reflection and transmission. These sharp band transmissions are the result of the control of the hundreds of interference layers with a relatively small difference in refractive index. This combination allows the creation of precise transitions with very little side lobe reflection, resulting in improved UV optical density with very low levels of visible color.

These UV filters can be designed to block up to about 420 nm, with very little yellowness, improving their applicability for ophthalmic devices. They can replace existing filters for applications such as projection systems, UV reduction and scintillator detectors.

The company says the filters allow a reduction in weight and size when incorporated into photonic products. They achieve more than 99 percent reflectivity at all angles and use the Giant Birefringent Optical design, which prevents any loss of reflectivity.

3M Co.
Optical Components Program
• Pulse Compressor •



The Pulse Compressor from 3M Co. of Austin, Texas, manages the chromatic dispersion that can limit transmission speeds in optical communications systems. Earlier solutions required the use of bulky modules with dispersion-compensating fiber, which has high optical attenuation and increased optical linear effects.

Suitable for metropolitan systems, the device corrects for high amounts of dispersion with low optical insertion loss, requiring fewer optical amplifiers. Subsea system designers can use it to match the large residual dispersion accumulated over thousands of kilometers of dispersion-mapped fiber trains.

The company says it developed several process technologies and materials to make the Pulse Compressor, including a fiber Bragg grating fabrication technique and special packaging to ensure stability and long-term reliability. The fiber Bragg grating is several meters long with a pitch accuracy of >4 pm. 3M also developed a diamond-like UV-transparent glass and a process whereby it can be deposited on the fiber. The material protects the fiber during grating inscription while maintaining the tight geometry tolerances the fiber needs for long-length-grating fabrication.

The grating period is linearly chirped to reflect lagging wavelengths before faster wavelengths that must travel farther into the grating before they are reflected. An optical circulator separates the light input from the output.